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The porous film which is superior in flexibility

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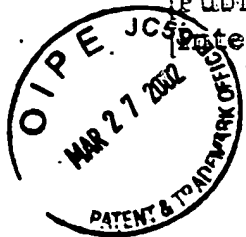
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[Title of the Invention] Hydrophilized Porous Film or Sheet

[Claim]

[Claim 1] A porous film or sheet which is obtained by uniaxially or biaxially stretching a film or sheet obtained by melt extrusion of a polyolefin resin composition containing at least a filler, and whose pore volume per 1cm³ unit volume of the film is 0.1 cc or more, and a hydrophilized porous film or sheet characterized by that moisture absorption is 0.1 wt% or more, moisture vapor permeability is 500 g/m² □24 hr or more, and yet the bending resistance both in lengthwise direction and in lateral direction of the film complies with the formula (I)

$$\text{Bending resistance} \square 0.193 \times \text{Film Thickness} + 35 \quad (\text{I})$$

Wherein the unit of bending resistance is mm, and the unit of film thickness is μm .

[Detailed Description of the Invention]

[Industrial Application Field]

The present invention relates to a hydrophilized porous film or sheet. More particularly, it relates to a porous film or sheet which is obtained by uniaxially or biaxially stretching a film or sheet formed by melt molding a polyolefin resin composition containing at least a filler, and which is excellent in moisture vapor permeability and moisture absorption, and is suitably used for packaging, battery separators, filter medium, medical application, and the like.

[Prior Art]

The present inventors have previously applied for patents regarding its manufacturing methods of a polyolefinic porous film or sheet containing fillers used for packagings, battery separators, filter medium, medical application, and the like (Japanese Patent Application No. S57-172598, (Publication Laid-open No. S59-62117), Japanese Patent Application No. S58-10232, (Publication Laid-open No. S59-136334), Japanese Patent Application No. S58-14937 (Publication Laid-open No. S59-140235).

[Problem to be Solved by the Invention]

However, although the porous film or sheet shows good moisture vapor permeability, it hardly shows the moisture absorption property, so that it is not preferable for use in the field such as battery separators and medical application where moisture absorption property is required.

[Means for Solving the Problem]

The present inventors reached to the present invention, as a result of performing a very hard study to provide a film or sheet excellent both in moisture permeability and moisture absorption property, as well as in flexibility.

Namely, the present invention provides a non-stretched film or sheet which is obtained by melt extrusion of a polyolefin resin composition containing at least a filler, and said non-stretched film or sheet is uniaxially or biaxially stretched to form a porous film or sheet whose pore volume per unit volume of 1cm³ of the film is 0.1 cc or more, and a hydrophilized porous film or sheet

characterized by that moisture absorption is 0.1 wt% or more, moisture vapor permeability is $500 \text{ cm}^2/24 \text{ hr}$ or more, and yet the bending resistance both in lengthwise direction and in lateral direction of the film complies with the formula (I)

$$\text{Bending resistance} \leq 0.193 \times \text{Film Thickness} + 35 \quad (\text{I})$$

Wherein the unit of bending resistance is mm, and the unit of film thickness is μm .

As for a polyolefin resin used by the present invention, high density polyethylene, medium density polyethylene, and linear low density polyethylene may be used independently, or as the mixture of two or more kinds of them, and further low density polyethylene produced by a high pressure process may be mixed. And, crystalline polypropylene is also used.

As for a filler, inorganic and organic fillers are used. Calcium carbonate, talc, clay, kaolin, silica, diatomite, magnesium carbonate, barium sulfate, calcium sulfate, aluminum hydroxide, zinc oxide, magnesium hydroxide, calcium oxide, magnesium oxide, titanium oxide, alumina, mica, asbestos powder, glass powder, volcanic glass balloon, zeolite, silicate clay, and the like are used as an inorganic filler, and cellulose powders such as wood flours and pulp powders and the like are used as an organic filler. These are used independently or as a mixture. An average particle diameter of the filler is preferably $30 \mu\text{m}$ or less, more preferably $10 \mu\text{m}$ or less, and most preferably $5 \mu\text{m}$ or less. When the particle size is too large, the density of pores in the stretched material may go down. The surface treatment of the filler is preferably performed with respect to dispersibility to the resin as well as stretchability, and a preferable result is given by treating with fatty acid or its metallic salt.

Although the porous film or sheet of the present invention consists fundamentally of a polyolefin resin and a filler, a hydrocarbon polymer in liquid type or wax type, for example, may be blended for keeping flexibility of the film or sheet. As for a hydrocarbon polymer in liquid type or wax type, liquid polybutadiene, liquid polybutene, liquid polyisoprene, and their derivatives are used. Among these, a hydroxyl terminated liquid polybutadiene and its derivative such as for example modified isocyanate, modified maleic anhydride, modified epoxy group and the like are used. Furthermore, a polyhydroxyl saturated hydrocarbon obtained by hydrogenating hydroxyl terminated liquid polybutadiene shows a good result.

Said polyhydroxyl saturated hydrocarbon is a hydrocarbon polymer in which main chain having at least 1.5 hydroxyl groups per molecule is saturated, or mostly saturated, and one having a number average molecular weight of $400 \sim 48000$, preferably within the range of $500 \sim 20000$ (by the vapor pressure method) is used. When the number average molecular weight is too small, weather resistance is not enough, and when the number average molecular weight is too large, handling becomes difficult due to lowering of flow pressure. The average numbers of hydroxyl groups per molecule are 1.5 or more, preferably 1.8 or more, and more preferably in the range of $2 \sim 5$. And it is preferable that the main chain and long branch are terminated with hydroxyls.

Such polyhydroxy saturated hydrocarbon is obtained by the well-known method, for example, by hydrogenating a butadiene liquid polymer obtained by radical polymerization of the butadiene or with its copolymerization monomer

using hydrogen peroxide or the like as a polymerization initiator. As for a copolymerization monomer, isoprene, chloroprene, styrene, methyl (meta) acrylate, methyl vinyl ether, etc. are given.

Hydrogenation is performed by an ordinary method by using a nickel catalyst (for example, reduced nickel, Raney nickel), a cobalt catalyst, a platinum catalyst, a palladium catalyst, a rhodium catalyst, a ruthenium catalysts, their mixtures or alloy catalysts. When using polyhydroxy saturated hydrocarbon, an epoxy group containing liquid organic compound such as liquid epoxy resin or epoxidized vegetable oil may be used together.

In addition, a thermal stabilizer, an ultraviolet absorber, an antistatic agent, a pigment, a fluorescer, etc may be added to the polyolefin resin according to the conventional method.

The blending ratio of the polyolefin resin and the filler is in the range of 50-250 parts by weight, preferably 25-400 parts by weight of fillers as per 100 parts by weight of polyolefin resin. When using an epoxy group containing organic compound, or a liquid or wax type hydrocarbon polymer, the liquid or wax type hydrocarbon polymer is in the range of 1-100 parts by weight, preferably 3-70 parts by weight as per 100 parts by weight of polyolefin resin, and the epoxy group containing organic compound is in the range of 0-100 parts by weight, preferably 0-70 parts by weight, and total amounts of liquid or wax type hydrocarbon polymer and epoxy group containing organic compound is in the range of 1-100 parts by weight, preferably 3-70 parts by weight. When the blending ratio of the filler is less than 25 parts by weight, pores are not sufficiently formed in the stretched film, and the poriferous degree becomes low. And, when the blending ratio of the filler exceeds 400 parts by weight, the kneading characteristic, the dispersibility, and the moldability of the film or the sheet are inferior.

When the amount of the liquid or wax type hydrocarbon polymer exceeds 100 parts by weight, characteristics of the polyolefin resin are sacrificed so that satisfactory kneadability, film moldability and stretchability of the film or the sheet cannot be secured.

As for molding of film or sheet, film or sheet formation is performed by using an ordinary molding device and in accordance with an ordinary molding method, and inflation molding by a circular die, T-die molding by a T-die, and the like are suitably employed. Its choice depends on subsequent stretching method.

That is, in the case of uniaxial stretching, a roll stretching is preferably used, but, a tubular stretching emphasizing machine direction (take-up direction) stretching can also be used. And, stretching is acceptably performed either by one step or multiple steps of two steps or more, the draw ratio is 1.2 times or more, preferably 1.5 times or more.

Next, the case of biaxial stretching is described.

The biaxial stretching can be performed in lengthwise direction and in lateral direction simultaneously or sequentially, and a draw ratio is at least 1.2 times or more.

And, in both uniaxial stretching and biaxial stretching, heat relaxation treatment can be employed after stretching to stabilize film. And, surface treatment such as publicly known corona treatment and flame treatment, can also be performed.

The porous film or sheet whose pore volume per unit volume of 1 cm³ of

the film is 0.1 cc or more, obtained by uniaxially or biaxially stretching the film or sheet molded by melt extrusion of the polyolefin in composition including at least a filler, is subject to hydrophilization processing.

As for a hydrophilization processing method, it is treated by either of a surfactant or a deliquescent inorganic salt, or both mixture. As a surfactant used for the processing, any one of nonionic surfactant, cationic surfactant or anionic surfactant is used, or two kinds or more of these surfactants are used together.

As for a nonionic surfactant, polyol fatty acid monoglyceride, polyoxyethylene fatty acid ester, polyoxyethylene sorbitan fatty acid ester, polyoxyethylene alkyl ether, polyoxyethylene alkyl aryl ether, polyoxyethylene alkyl ether phosphate, and the like are used.

As for a cationic surfactant, quaternary ammonium salt, polyoxyethylene alkylamine, alkyl amine oxide, and the like are used. As for an anionic surfactant, alkyl sulfonic acid salt, alkyl benzene sulfonic acid salt, alkyl naphthalene sulfonic acid salt, alkylsulfo succinic acid salt, alkyl sulfonic acid ester acid, polyoxyethylene alkyl sulfonic acid ester salt, polyoxyethylene alkylaryl sulfonic acid ester salt, alkyl phosphate, polyoxyethylene alkyl phosphate, and the like are used.

And, as for a deliquescent mineral salt used for processing, the I group and II group metal halide of the periodic table are preferable, and particularly lithium chloride, calcium chloride, magnesium chloride, zinc chloride, and the like are used. Among these, a halide of alkali metal or alkaline earth metal are preferable especially. Two or more kinds of these deliquescent mineral salt may be used together.

When the porous film or the sheet is treated with either of a surfactant and a deliquescent mineral salt, or both mixture, dip coating, atomization, and the like are exemplified. However, a dip coating is conveniently used, where processing agent is mixed or dissolved in a liquid substantially mixed uniformly with the processing agent consisting of either of a simple surfactant, a deliquescent mineral salt, or both mixture, and the porous film or the sheet is dipped into the solution to impregnate the processing agent into the film or the sheet. As for a liquid substantially mixed uniformly with the surfactant, pure water and further alcohols such as methyl alcohol, ethyl alcohol and isopropyl alcohol are mentioned. And these mixtures may also be used. Furthermore, aliphatic hydrocarbon, aromatic hydrocarbon, chloroform, acetone, carbon tetrachloride, etc. may be used, or further these mixtures may also be used.

As for a liquid substantially mixed uniformly with the deliquescent mineral salt, alcohols such as methyl alcohol, ethyl alcohol, etc., acetone, diethyl ether, chloroform, etc., and further the mixture of pure water and said liquid which is mixed uniformly with pure water may also be used.

As for a liquid which is mixed substantially uniformly with the mixture of the surfactant and the deliquescent mineral salt, the pure water, and further alcohols such as methyl alcohol, ethyl alcohol, etc., and chloroform, acetone, etc. are mentioned. Or these mixtures may also be used further.

A concentration of surfactant is 0.1 wt% or more, and more preferably 1 wt% or more, and a concentration of deliquescent mineral salt is 0.05wt% or more, and preferably 0.1wt% or more.

When using the mixture of a surfactant and a deliquescent mineral salt,

the concentration of the surfactant is 0.1 wt% or more, and preferably 1wt% or more, and the concentration of the deliquescent mineral salt is 0.05wt% or more, and preferably 0.1wt% or more.

When the concentration of the surfactant is less than 0.1 wt% or the concentration of the deliquescent mineral salt is less than 0.05 wt%, hydrophilization processing is possible, but, it is not preferable since improvement effect is small. Hydrophilization processing may also be applied to the porous film or the sheet after subjecting to surface treatment by publicly known corona treatment and flame processing, etc. The film of the present invention has a moisture vapor permeability, moisture absorption, and flexibility equal to or more than a prescribed level. The moisture vapor permeability is $500 \text{ g/m}^2 \times 24 \text{ hr}$ or more, and preferably $1000 \text{ g/m}^2 \times 24 \text{ hr}$ or more, the moisture absorption is 0.1 wt% or more, and bending resistance both in lengthwise direction and in lateral direction of the film satisfies the formula (I).

$$\text{Bending resistance} \leq 0.193 \times \text{film thickness} \leq 35 \quad (\text{I})$$

Wherein the unit of bending resistance is mm and the unit of film thickness is μm .

Films which satisfy these requirements can be suitably used for battery separators, medical application, etc., and moisture vapor permeability and the moisture absorption can be selected according to the requirements within the scope explained in detail in the present invention.

[Examples]

Then, the present inventions is explained more precisely by examples, but it is not limited to the following examples, unless it is beyond the scope of the invention.

[Reference Example 1] (Production of polyhydroxy saturated hydrocarbon)

Into a 10L autoclave, 3kg of commercial liquid polybutadiene (by Nippon Soda; G-2000, molecular weight: 2000), 3 kg of cyclohexane, 300 g of carbon supported carrying ruthenium (5%) catalytic (by Japan Engelhard) were charged. The system was purged with purified argon gas, and then high purity hydrogen gas was supplied to the autoclave, and heating was started simultaneously to bring to a stable condition (internal temperature: approx. 100°C , and internal pressure: approx. 50 kg/cm^2) taking approx. 30 minutes. The condition was maintained for approx. 15 hours, and subsequently a hydrogenation reaction was stopped. The resulting polymer was liquid polyhydroxy saturated hydrocarbon having 5 g/100g of iodine number and 44 KOHmg/g of hydroxyl value.

[Example 1]

At first, 3.4 kg of a linear low density polyethylene resin (NOVATEC-U, FW20G made by Mitsubishi Chemical Industries) whose melt index was 1.0 g/10 min. and density was 0.918 g/cm^3 , and 5.8 kg of calcium carbonate (average particle diameter: $0.9 \mu\text{m}$, fatty acid treated) were stirred and mixed in a Henschel mixer, and then 0.8 kg of polyhydroxy saturated hydrocarbons obtained in Reference Example 1 was added to the mixture, and further stirred and mixed to obtain 10 kg of mixture.

This blending operation was repeated 10 times to finally obtain 100 kg of

mixture.

In addition, melt Perex is an extruded amount of resin in gram at 190°C and 2.16kg load according to ASTM D1238-70, and density is determined by using a density-gradient tube method at 20°C based on ASTM D1505.

The mixture obtained in this manner was mixed by a twin-shaft kneader DSM-65 (The Japan Steel Works, Ltd.) and then pelletized. The pellets are subject to inflation molding by using a 50 mmφ extruder to form 120μm thickness film.

Herein, the extrusion conditions are the following:

Cylinder temperature: 170-190-190°C

Head and Die temperature: 190-190°C

Blow ratio: 2.5

The film obtained in such a manner was uniaxially stretched by a roll stretching machine, and then heat treated to form 80μm thickness porous film having a pore volume of 0.27 cc per unit volume of 1 cm³.

Stretching and heat relaxation treatment condition are the following:

Stretching temperature: 70 °C

Draw ratio: 3.0

Heat treatment temperature: 100 °C

Heat relaxation rate: 10%

The porous film was dipped into the solution of a prescribed concentration in which either of alkyl naphthalene sulfonic acid soda (Perex NB paste by Kao) of an anionic surfactant or lithium chloride, or a mixture of them at a prescribed ratio was dissolved in either of ethyl alcohol or pure water or a mixture of them at a prescribed ratio for a prescribed period and then was air-dried after squeezed by rollers.

The moisture vapor permeability, moisture absorption and bending resistance of this treated film were shown in Table-1, and it was thus understood that the moisture absorption was extremely improved compared with Comparative Example 1.

In addition, the measuring methods are the following:

- 1) Pore volume (cc/cm³): is measured by using a mercury porosimeter (by AMINCO, 60,000 psi porosimeter). The pore volume was expressed by cumulative pore volume ranging from micro-pore to 10μm in radius per 1cm³ unit volume of the film on the cumulative pore volume distribution chart.
- 2) Moisture vapor permeation rate (g/m²·24 hr): It was determined at 30°C and 90% relative humidity(R.H.) according to JIS Z 0208-1976.
- 3) Moisture absorption (weight %): After drying the film in a desiccator with calcium chloride for 24 hours (in a thermostatic chamber at 20°C), the increase in the weight exposed at 20°C and 65%(R.H.) for 24 hours was measured in wt.%.
- 4) Bending resistance (mm) : it was measured by the 45°cantilever method according to JIS L 1018-1977. The measurement was performed at 20°C and 65%(R.H.)

[Comparative Example 1]

It was a porous film with a thickness of 80μm obtained by uniaxial stretching by a roll stretching machine and without hydrophilization treatment.

The measuring results were shown in Table-1, and it was indicated that although the moisture vapor permeability was suitably high, and the bending

resistance was suitably low, the moisture absorption was so low that the moisture absorption property was hardly shown, or only an insufficient moisture absorption property was shown.

[Comparative Example 2]

The porous film having a film thickness of 80 μ m obtained by uniaxial stretching by a roll stretching machine in Example 1 was dipped into a 5 wt% aqueous solution of lithium chloride for 10 seconds, and then air-dried as the same manner as stated in Example 1. The measuring results were shown in Table-1. The moisture absorption was only 0.009 wt% and it was insufficient in moisture absorption property.

[Comparative Example 3]

The porous film having a film thickness of 80 μ m obtained by uniaxial stretching by a roll stretching machine in Example 1 was dipped into a 0.05 wt% aqueous solution of alkyl naphthalene sulfonic acid soda used in Example 1 for 10 seconds, and then air-dried as the same manner as stated in Example 1. After dipping the porous film having a film thickness of 80 μ m obtained by uniaxial stretching by a roll stretching machine in Example 1 into a 0.05 wt% aqueous solution of used in Example 1 for 10 seconds, it was air-dried similarly to Example 1. The measuring results were shown in Table-1. The moisture absorption was 0.05 wt% and it was insufficient in moisture absorption property.

[Example 2]

At first, 3.9 kg of the same linear low density polyethylene resin as used in Example 1, 0.2 kg of high-pressure low density polyethylene whose density is 0.924 g/cm³ and melt index is 2.0 g/10 minutes (NOVATEC-L, F150 made by Mitsubishi Chemical Industries) and 5.2 kg of the same calcium carbonate as used in Example 1 are stirred and mixed in a Henschel mixer. Then, 0.6 kg of polyhydroxy saturated hydrocarbons obtained in Reference Example 1 and 0.1 kg of epoxidized soybean oil (ADK Cizer 0-130L made by Adeka Argus) are added, stirred and mixed furthermore, and kneading and pelletization were performed by completely the same method as stated in Example 1, and then inflation film forming was performed at the same conditions as in Example 1 to obtain film with thickness of 120 μ m. The said film was then subject to uniaxial stretching and heat relaxation treatment by using a roll stretching machine. Stretching and heat relaxation conditions are the following:

Stretching temperature: 70 \square

Draw magnification: 2.0

Heat relaxation temperature: 100 \square

Heat relaxation rate: 13%

Then, said uniaxial stretched film was stretched in lateral direction by a tenter, and furthermore, heat relaxation treating was performed in lateral direction. The conditions are the following:

Lateral stretching temperature: 95 \square

Lateral draw ratio: 2.5

Lateral heat relaxation temperature: 105 \square

Heat relaxation rate in lateral direction: 15%

The film obtained in this way was a porous film having a film thickness of

75 μ m and a pore volume of 0.36 cc per unit volume 1cm³ of film.

Said porous film was then dipped into the solution at a prescribed concentration in which either of alkyl naphthalene sulfonic acid soda or lithium chloride, or a mixture of them at a prescribed ratio was dissolved in either of ethyl alcohol or pure water or a mixture of them at a prescribed ratio for a prescribed period and then was air-dried in the same manner as stated in Example 1.

The moisture vapor permeability, moisture absorption, and bending resistance of this treated film were shown in Table-2.

[Comparative Example 4]

It was a porous film having film thickness of 75 μ m obtained by lateral stretching by a tenter in Example 2. The measuring result were shown in Table-2.

[Example 3]

Except that the linear low density polyethylene in Example 1 was replaced with a high density polyethylene (NOVATEC, ES300 by Mitsubishi Chemical Industries) whose melt index was 0.04 g/10 min. and density was 0.960 g/cm³, the same inflation molding as stated in Example 1 was performed to form a film with a thickness of 120 μ m. Then, the said film was subject to uniaxial stretching by using a roll stretching machine to form a porous film whose film thickness was 72 μ m and pore volume per unit volume 1cm³ of the film was 0.26 cc. Stretching conditions were the same as in Example 1.

The said porous film was dipped into either of polyoxyethylene sorbitan monolaurate (Reodol TW-L120 by Kao) of a nonionic surfactant or lithium chloride, or a mixture of them at a prescribed ratio was dissolved in either of ethyl alcohol or pure water or a mixture of them at a prescribed ratio for a prescribed period and then was air-dried in the same manner as stated in Example 1.

The moisture vapor permeability, moisture absorption, and bending resistance of this treated film were shown in Table-3.

[Comparative Example 5]

It was a porous film having a film thickness of 72 μ m obtained by roll stretching in Example 3.

The measuring results were shown in Table-3.

[Comparative Example 6]

The film with a thickness of 72 μ m obtained by uniaxial stretching by a roll stretching machine in Comparative Example 3 was dipped in 0.03 wt% ethyl alcohol solution of lithium chloride for 10 seconds, and air-dried in the same manner as stated in Example 1. The measuring results were shown in Table-3. Although the effect of hydrophilization treatment was observed for the moisture absorption of 0.07 wt% compared with Comparative Example 5, the moisture absorption was low and still unsatisfactory.

[Effects of the Invention]

The film or sheet of the present invention is excellent in the moisture vapor permeability, moisture absorption and flexibility, and very effectively

used as a material for packaging, battery separators, filter medium, medical application and so on.

Table-1

	Surfactant		Deliquescent inorganic salt		Solvent		Dipping time (sec)	moisture Vapor Permeability (g/m ² □24hr)	Moisture Absorption property (wt%)	Bending resistance	
	Substance	Concentration (wt%)	Substance	Concentration (wt%)	Ethyl alcohol (vol.%)	Pure Water (vol.%)				Lengthwise (mm)	Lateral (mm)
Example 1	Alkyl naphthalene sulfonic soda	3	□	□	0	100	10	3200	0.16	25	28
	□	□	Lithium chloride	5	50	50	10	3300	4.4	25	27
C.E.* 1	Alkyl naphthalene sulfonic soda	3	Lithium chloride	1	0	100	10	3100	0.54	26	28
	□	□	□	□	□	□	□	4000	0.02	25	28
C.E.* 2	□	□	Lithium chloride	5	0	100	10	3900	0.04	25	28
	□	□	□	□	□	□	□	3800	0.05	25	28
C.E.* 3	Alkyl naphthalene sulfonic soda	0.05	□	□	0	100	10				

C.E.*: Comparative Example

Table-2

	Surfactant		Deliquescent inorganic salt		Solvent		Dipping time (sec)	Moisture Vapor Permeability (g/m ² □24hr)	Moisture Absorption property (wt%)	Bending resistance	
	Substance	Concentration (wt%)	Substance	Concentration (wt%)	Ethyl alcohol (vol.%)	Pure Water (vol.%)				Lengthwise (mm)	Lateral (mm)
Example 2	Alkyl naphthalene sulfonic soda	6	□	□	0	100	10	4600	0.20	29	31
	□	□	Lithium chloride	3	100	0	10	4800	2.7	30	31
	Alkyl naphthalene sulfonic soda	3	Lithium chloride	3	0	100	10	4300	1.8	30	30
C.E.* 4	□	□	□	□	□	□	□	8000	0.04	31	29

C.E.*: Comparative Example

Table-3

	Surfactant		Deliquescent inorganic salt		Solvent		Dipping time (sec)	Moisture Vapor Permeability (g/m ² □24hr)	Moisture Absorption property (wt%)	Bending resistance	
	Substance	Concentration (wt%)	Substance	Concentration (wt%)	Ethyl alcohol (vol.%)	Pure Water (vol.%)				Lengthwise (mm)	Lateral (mm)
Example 2	Polyoxyethylene sorbitan monolaurate	3	Lithium chloride	5	0	100	10	3800	4.6	35	37
	□	□	Lithium chloride	1	100	0	10	3900	0.89	36	34
C.E.* 5	□	□	□	□	□	□	□	4000	0.02	36	36
C.E.* 6	□	□	Lithium chloride	0.03	100	0	10	4000	0.07	37	36

C.E.*: Comparative Example